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Measurement of the thermal diffusivity of a silica fiber bundle using a laser and an IR camera

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Résumé. (A3600GV) We propose a lightweight method for the determination of heat diffusivity of silica fiber bundles based on the use of a laser and an IR camera. The fiber bundle is maintained in traction in a holder; exposition is made as a step function, followed by a laser shutdown. The movie obtained by the IR camera is then processed : frame averaging, background computation and subtraction, image smoothing, extraction of the IR signal along the fiber bundle. A 1D model has been developed. This problem admits an analytic solution that we have obtained through the use of Laplace transforms. Several identification methods are proposed and tested, and have been compared favorably with an existing method based on periodic heating. Results are in agreement with literature values.

1. Introduction

The determination of fiber thermal properties is a key issue when designing composite materials with thermal and/or thermostructural applications. Many methods exist for carbon fibers, which are based on their electrical conductivity [1]; not all of them are applicable to insulating materials such as glass or silica fibers. Photothermal microscopy [2] has been investigated on *C/C* composites [3] and *in-situ* characterization of a carbon fiber thermal diffusivity was made. Photoacoustics [4] and thermal flash under microscope [5] have been used for the measurement of glass fiber heat diffusivity. Measurements in periodic regime have been proposed for carbon and tungsten fibers at high temperatures, using a laser source and a chopper, and an IR detector [6]. The thermal diffusivity was obtained from the evolution of the phase lag and of the amplitude as the distance from the excitation point increases. We propose here an alternative, lightweight method, based on the use of a laser and an IR camera, and using a step excitation instead of a periodic signal. The fibers are excited by the laser, and they lose heat with respect to the surroundings; nonetheless, the heat loss coefficient is identified together with the thermal diffusivity. In the following, details of the experimental procedure and of the employed model are given, then, various identification procedures are tested and compared.

2. Experimental

The characterized fibers are silica fibers with density $\rho \approx 2.21 \text{ g.cm}^{-3}$ extracted from a commercial fabric. The fiber bundle of diameter 0.45 mm is maintained in traction in a holder ; it is painted in black, and it has been checked by scanning electron microscopy that the paint thickness was less than $0.3 \text{ }\mu\text{m}$, so that its impact on the effective thermal diffusivity is negligible. In order to minimize the system perturbation due to uncontrolled heat losses by natural or forced convection, the setup is placed in a vacuum cell, in which the pressure is lowered down to 10^{-3} Pa . A 30 mW laser is used at $1/3$ full power. Exposition is made as a step function. After reaching steady state, the power is cut off, and the thermal relaxation is also recorded. An FLIR SC7000 IR camera with cooled detector is used for the continuous recording of the signal at 50 Hz frequency. The camera pixel size was recovered by *in-situ* calibration using the dimensions of the sample holder. The movie is then processed using FLIR proprietary software and ImageJ freeware : frame averaging, computation and subtraction of the background, and image smoothing. Time evolution of the temperature on selected points, as well as thermal profiles at selected instants have been extracted for identification of thermophysical data, using the model presented in the next part.

3. Model

An approximate model of the experiment may be conveniently derived and exploited. The hypotheses of the model are :

- 1D geometry. Actually, the bundle thickness was less than 3 pixels wide.
- A constant heat flux Φ_0 is injected at the spot center, considered as a single point.
- The bundle is considered symmetrical with respect to the spot.
- Heat losses on the wire are considered as having a constant transfer coefficient h .
- Conductivity λ and heat capacity c_p are considered constant.

Let θ be the excess temperature with respect to ambient. The governing equation is :

$$\rho c_p \frac{\partial \theta}{\partial t} - \lambda \frac{\partial^2 \theta}{\partial x^2} + \frac{4h}{D} \theta = 0 \quad (1)$$

where D is the fiber bundle diameter.

It may be fully solved both in the case of heating and of cooling. For the heating period, the boundary and initial conditions are the following :

$$-\lambda \frac{\partial \theta}{\partial x} \Big|_{x=0} = \Phi_0 \quad (2)$$

$$\lim_{x \rightarrow \infty} \theta(x, t) = 0 \quad (3)$$

$$\theta(x \neq 0, t = 0) = 0 \quad (4)$$

This problem admits a solution, that we have obtained through the use of Laplace transforms.

$$\theta(x, t) = \frac{\Phi_0 \sqrt{a}}{2\lambda \sqrt{b}} \left[\exp \left(-\sqrt{\frac{bx^2}{a}} \right) \operatorname{erfc} \left(\sqrt{\frac{x^2}{4at}} - \sqrt{bt} \right) - \exp \left(\sqrt{\frac{bx^2}{a}} \right) \operatorname{erfc} \left(\sqrt{\frac{x^2}{4at}} + \sqrt{bt} \right) \right] \quad (5)$$

where $a = \frac{\lambda}{\rho c_p}$ is the thermal diffusivity and $b = \frac{4h}{\rho c_p D}$ is the scaled heat loss coefficient.

Several particular cases are of special interest, as discussed below.

3.1. Large x approximation

In the case where $\varepsilon = \sqrt{bt}$ is small with respect to $x/(2\sqrt{at}) = \xi$, the "erfc" functions terms in eq. (5) may be approximated using a 1st order series development ($\text{erfc}(\xi + \varepsilon) \approx \text{erfc}(\xi) + \frac{2\varepsilon}{\sqrt{\pi}} \exp(-\xi^2) + \mathcal{O}(\xi^4)$). So, considering sufficiently large x while keeping adequate time values ($t \ll x/(2\sqrt{ab})$) yields the following approximation :

$$\ln(\theta) \approx -\frac{x^2}{4at} + K_0 + K_1(t) - 2\ln(|x|) + \frac{K_2(t)}{x^2} + \mathcal{O}\left(\frac{1}{x^4}\right) \quad (6)$$

where K_i are constants or functions of time only. So, since

$$\frac{\partial \ln \theta}{\partial (x^2)} \approx -\frac{1}{4at} \quad (7)$$

we may identify a by plotting the logarithm of the images $\ln \theta(x, t)$ versus x^2 and fit it with straight lines for selected time values.

3.2. Steady-state profile and relaxation

The limit of Eq. 5 when $t \rightarrow \infty$ is :

$$\theta(x, \infty) = \frac{\Phi_0 \sqrt{a}}{\lambda \sqrt{b}} \exp\left(-\sqrt{\frac{bx^2}{a}}\right) \quad (8)$$

Consequently, the ratio $\sqrt{b/a}$ may be straightforwardly identified as the slope of $-\ln(\theta) = f(|x|)$. This only has an interest if we are able to identify independently the b coefficient. Indeed, this is the case, since it is possible to solve the heat equation for the cooling case. The established steady-state thermal profile Eq. 8 is taken as initial condition, and the injected power Φ_0 is cancelled. The initial and boundary conditions are :

$$-\lambda \frac{\partial \theta}{\partial x} \Big|_{x=0} = 0 \quad (9)$$

$$\lim_{x \rightarrow \infty} \theta(x, t) = 0 \quad (10)$$

$$\theta(x \neq 0, t = 0) = \frac{\Phi_0 \sqrt{a}}{\lambda \sqrt{b}} \exp\left(-\sqrt{\frac{bx^2}{a}}\right) \quad (11)$$

The solution is :

$$\theta(x, t) = \frac{\Phi_0 \sqrt{a}}{\lambda \sqrt{b}} \exp\left(-\sqrt{\frac{bx^2}{a}}\right) \text{erfc}(\sqrt{bt}) \quad (12)$$

which means that the thermal profile is decreasing exponentially with time, while keeping exactly the same shape. For large enough values of t , one has $\ln(\theta) \approx K - bt - 1/2 \ln(\pi bt)$, where K is a constant. The coefficient b may thus be identified by a plot of $\ln(\theta) = f(t)$ for any point chosen on the thermal profile. Having in hand the value of b , the thermal diffusivity may be recovered from the knowledge of the characteristic length $\sqrt{a/b}$ taken in the steady-state or during cooling.

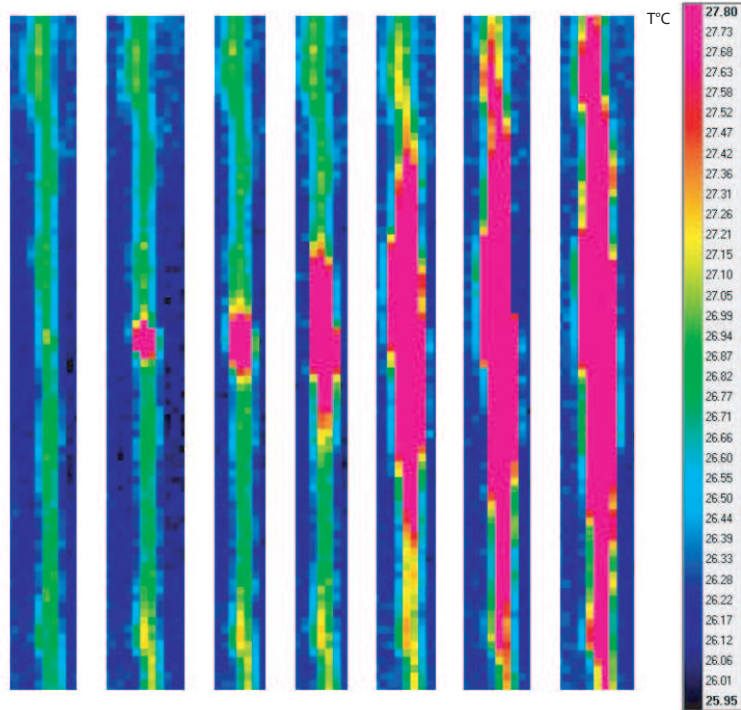


Figure 1. Example of recorded thermal images at initial state $t_0 - \epsilon$, and $t_0 + 0.2$ s, $+0.6$ s, $+2.0$ s, $+10.0$ s, $+20.0$ s, and $+60.0$ s from left to right. The temperature scale is expressed in degrees Celsius.

4. Some results

Tests were performed on a bundle containing *ca.* 300 fibers. Its diameter was roughly $450 \mu\text{m}$. Figure 1 is an example of recorded thermal images. The pixel size was $150 \mu\text{m}$. We can see that the bundle was only roughly 3 pixels wide, thus confirming the 1D approximation in the model.

The data was smoothed with respect to time using a 5-frame ($1/10$ s) average filter. The background noise was recorded during one second before the start of the experiment, *i.e.* 50 frames; its average was subtracted to all the posterior frames for the preparation of the scaled data θ . Figure 2 is a plot of $\ln(\theta)$ *vs.* time and position.

We have performed identification using the "large x , small t " profile on the one hand, and using the "steady state and relaxation" procedure on the other hand. Fig. 3 a) shows plots of $\ln \theta = f(x^2)$ for selected time values. We see that there is a linear part for sufficiently large x^2 . The inverse of the slope is plotted against t , as shown in fig. 3 b), to obtain directly the diffusion coefficient a .

Figs. 4 are curves of $\ln(\theta)$ *vs.* x in steady state and *vs.* t during relaxation. We can see that there is a good agreement with the model equations. Parameters a and b are recovered from the slopes of these curves.

The estimates of the coefficients a and b have been used as a first guess for a full nonlinear fit of Eq. (5) using the *ad-hoc* Levenberg-Marquardt routine in the Gnuplot freeware [8], as shown in Fig. 2b). Finally, we have used the periodic heating method described in [6] for another distinct measurement of the thermal diffusivity, from the space variation of the phase lag and of the logarithm of the amplitude.

All results are collected in Table 1.

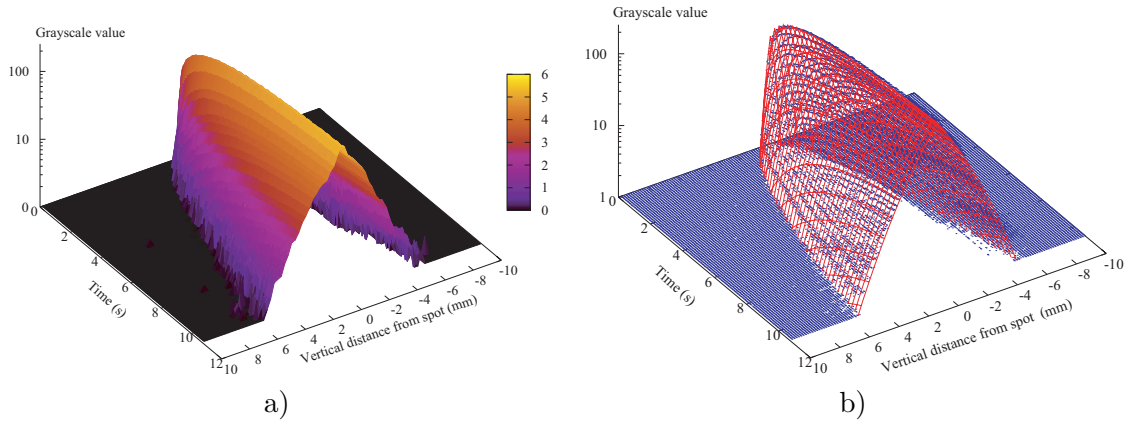


Figure 2. a) Plot of the reduced temperature *vs.* time and position. b) Same as a), with a fitted surface from Eq. (5)

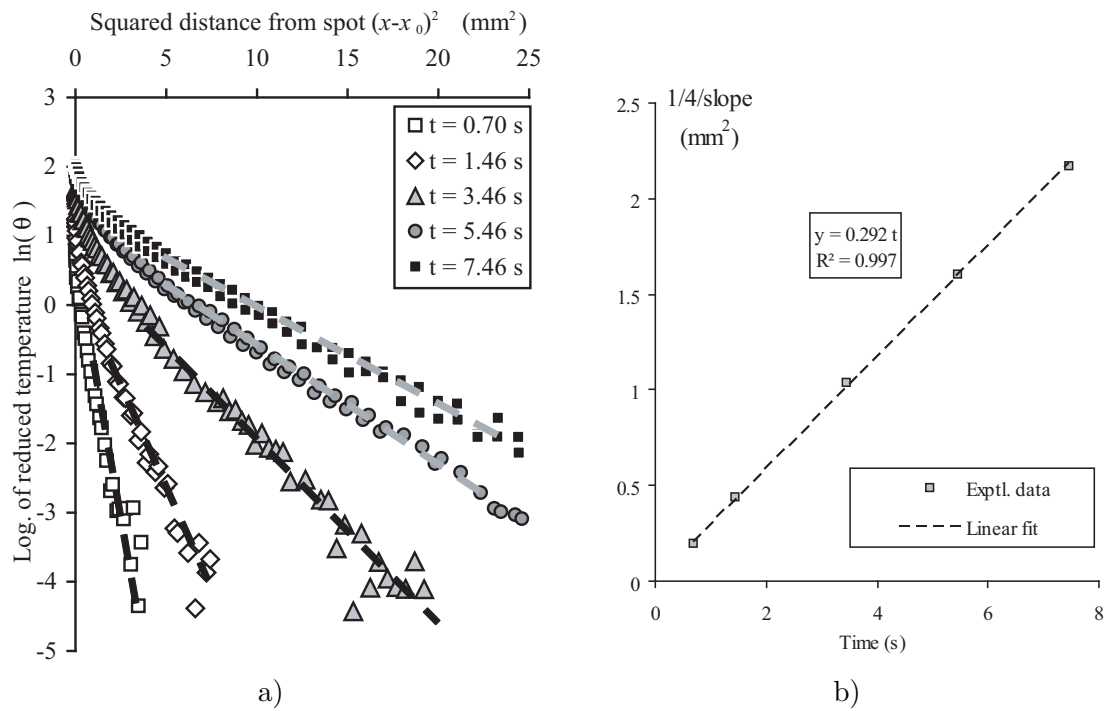


Figure 3. a) Curves of $\ln \theta = f(x^2)$ at several times. b) Inverse of the slopes *vs.* time.

Table 1. Summary of identified values of the thermal diffusivity in $mm^2 \cdot s^{-1}$.

	Method	Diffusivity	\pm	Error
	"Large x , small t "	0.29	\pm	$1 \cdot 10^{-2}$
	"Steady-state + relaxation"	0.31	\pm	$2 \cdot 10^{-2}$
	Direct adjustment	0.236	\pm	$7 \cdot 10^{-4}$
	Periodic heating	0.241	\pm	$5 \cdot 10^{-3}$

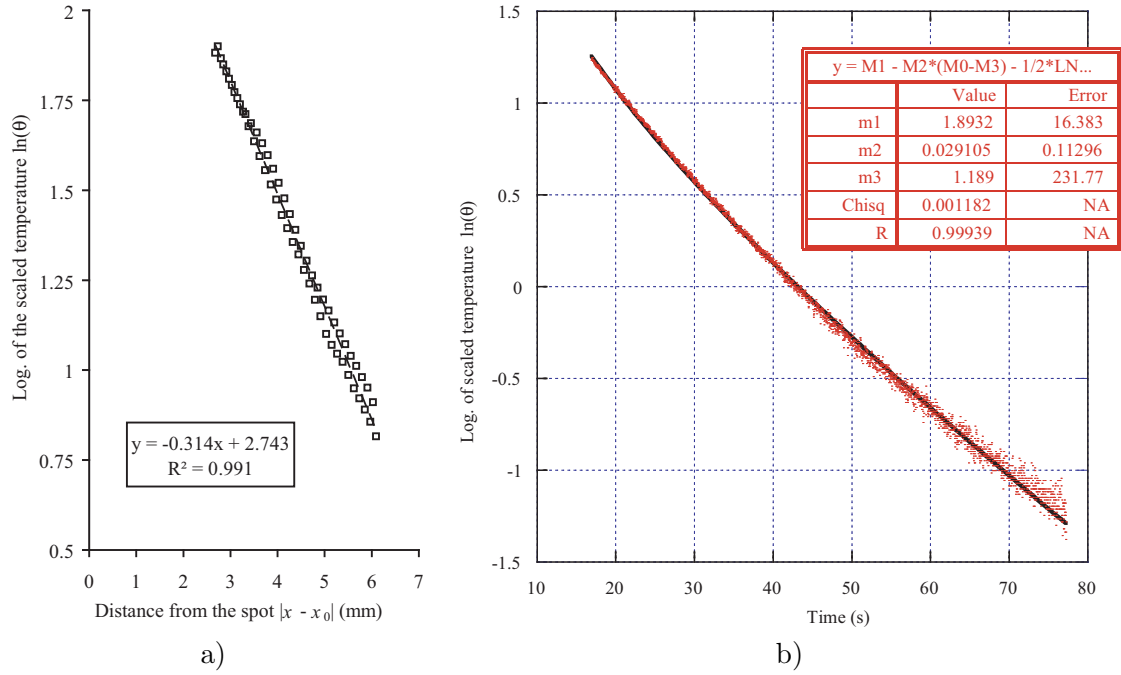


Figure 4. Curves of a) $\ln \theta = f(x)$ at steady state and b) relaxation : $\ln \theta$ vs. time after laser shutdown.

We can see that the first two methods, though simpler to use, are in fair agreement with the last two. However there is a slight over-estimation for the former ones. This can arise from several factors. First, the estimation of the slopes of $\ln \theta$ vs. x^2 is performed in the region of the profiles where the noise is highest, which hampers somewhat the accuracy of the determination. Second, the estimation of the slope of $\ln \theta$ vs. $|x|$ is performed at the last time frame before shutting down the laser, which is not exactly the steady state.

The value found for the thermal diffusivity is lower than those given by [9] (values ranging between 0.3 and $0.9 \text{ mm}^2.\text{s}^{-1}$) but higher than those given by [5] ($0.12 \text{ mm}^2.\text{s}^{-1}$). The heat capacity has also been measured by DSC, giving a value of $1.5 \text{ J.g}^{-1}.\text{K}^{-1}$. So, the heat conductivity at room temperature is evaluated at $0.86 \text{ W.m}^{-1}.\text{K}^{-1}$, in agreement with many literature values for silica-rich glasses.

5. Conclusion

We have developed a lightweight method for the measurement of thermal diffusivity of glass fibers using a laser and an IR camera. A step heating followed by simple cooling were used, in contrast with preceding methods based on periodic heating. Several identification procedures were developed and tested, with a good agreement between each other and with respect to periodic heating tests. The interest of the non-periodic method is its efficiency : the experimental manipulation is very simple, and the identification using the steady-state profile and the relaxation time is very practical. Moreover, since one works with the logarithm of the signal, no absolute thermal or calorimetric calibration is necessary. This work may be of importance in the prediction of effective thermophysical properties for composite materials, in conjunction with the determination of the matrix properties, though the latter can be attained more easily, *e.g.* through the classical flash method. Future work will be dedicated to this prediction strategy.

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